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[Title of the Invention]	**Gasket Material and Gasket Utilizing Same**
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[Title of the Invention]   Gasket Material and Gasket Utilizing Same

[Claims]

[Claim 1]   A gasket material, composed of a laminated sheet obtained by the lamination of a biaxially extended porous polytetrafluoroethylene film consisting of nodes and of fibrils that connect said nodes, wherein said gasket material is characterized in that

said biaxially extended porous polytetrafluoroethylene film is essentially free of nodes whose diameter or major axis exceeds 3 µm per scanning electron microscope observation area of 330 μm^2.

[Claim 2]   A gasket material as defined in Claim 1, wherein the elongation at break is 200% or less, as determined by the test defined in JIS K7113.

[Claim 3]   A gasket material as defined in Claim 1 or 2, wherein the tensile strength of the matrix of the gasket material is 10 kgf/mm^2 or greater.

[Claim 4]   A gasket material as defined in any of Claims 1 through 3, wherein a gasket material with a thickness of 3 mm or greater and a porosity of 75% or lower has a stress relaxation ratio of 35% or lower, as determined by a 96-hour ATRS test at 93°C.

[Claim 5]   A gasket material as defined in any of Claims 1 through 4, wherein said laminated sheet contains at least one interposed reinforcing layer composed of a material that is more rigid than said extended porous polytetrafluoroethylene film.

[Claim 6]   A gasket material as defined in Claim 5, wherein said reinforcing layer is obtained by squeezing the pores of the extended porous polytetrafluoroethylene film.

[Claim 7]   A gasket, obtained by forming an appropriate shape from a gasket material as defined in any of Claims 1 through 6.

[Detailed Description of the Invention]
[0001]
[Field of Industrial Utilization]

The present invention relates to a gasket for sealing piping flanges, shaft or pin lubrication points, or the like, and to a method for manufacturing the material for such a gasket. In particular, [the present invention] relates to an extended porous polytetrafluoroethylene gasket material that is highly resistant to creep and is suitable for parts fastened with considerable fastening force, and to a gasket in which this material is used.

[0002]
[Prior Art]

Gaskets highly resistant to corrosion and heat are used for the joints of piping systems carrying pharmaceuticals, foodstuffs, and other corrosive fluids. Because of their excellent corrosion resistance, heat resistance, and adhesion to fastening surfaces, gaskets made of extended porous polytetrafluoroethylene (hereinafter abbreviated as "ePTFE") have attracted attention in recent years. For example, Japanese Laid-Open Utility Model Application 3-89133 describes annular gaskets manufactured from ePTFE by a process in which a sheet obtained by laminating and integrating ePTFE films in a prescribed thickness is punched out to produce annular shapes.

[0003]

Here, the term "ePTFE film" refers to a porous polytetrafluoroethylene (PTFE) film fashioned into a fibrous structure by stretching. In the method for manufacturing such a film, a molding aid is commonly removed from a paste-molded article obtained by mixing the molding aid with a fine PTFE powder (crystallinity: 90% or higher), and the article is then stretched at a high temperature (about 300°C) below the melting point of PTFE (about 327°C) and calcined as needed, as described in Japanese Patent Publication 51-18991. In ePTFE obtained by biaxial stretching, fibrils 1 (linear molecular bundles unraveled and drawn out by the stretching of folded crystals) spread out radially away from nodes 2 composed of folded crystals, forming a spiderweb, fibrous structure in which the nodes 2 that connect the fibrils 1 form isolated islands, and a large number of spaces are formed by the fibrils 1 and the nodes 2, as shown in Figure 9. Spaces between the fibrils or between the fibrils and nodes serve as pores 3.

[0004]

[Problems Which the Invention Is Intended to Solve]

When, however, an ePTFE gasket fabricated using a gasket material composed of an ePTFE film thus manufactured is used for a long time while remaining fastened at considerable pressure, the gasket is deformed (subjected to creep), and, as a result, the sealing properties are adversely affected and the gasket ultimately loses its ability to function. In other words, the time (hereinafter referred to as "gasket life") elapsed before the gasket loses its ability to function is too short.

[0005]

In view of this situation, it is an object of the present invention to provide an ePTFE gasket characterized by better creep resistance than in the past, and to provide a gasket material that allows such a gasket to be obtained.

[0006]

[Means Used to Solve the Above-Mentioned Problems]

The inventors discovered that creep in ePTFE gaskets is caused by nodes, i.e., that fibrils in which the folded crystals have been adequately drawn out by stretching withstand deformation by the compressive force during use, but the nodes composed of inadequately stretched folded crystals are deformed by the compressive force during use, bringing about creep and ultimately causing the gasket to lose its ability to function. In view of this, the inventors perfected the present invention after focusing on the use of ePTFE films with very small nodes in which virtually all folded crystals have been unraveled.

[0007]

Specifically, the gasket material of the present invention is composed of a laminated sheet obtained by the lamination of a biaxially extended porous polytetrafluoroethylene film consisting of nodes and of fibrils that connect these nodes, wherein this gasket material is characterized in that the aforementioned biaxially extended porous polytetrafluoroethylene film is essentially free of nodes whose diameter or major axis exceeds 3 μm per scanning electron microscope observation area, of 330 μm^2. It is preferable for the elongation at break to be 200% or less, as determined by the test defined in JIS K7113, and for the tensile strength of the matrix

of the gasket material to be 10 kgf/mm^2 or greater. It is also preferable for a gasket material with a thickness of 3 mm or greater and a porosity of 75% or lower to have a stress relaxation ratio of 35% or lower, as determined by a 96-hour ATRS test at 93°C.

[0008]

Moreover, it is preferable for the aforementioned laminated sheet to contain at least one interposed reinforcing layer composed of a material that is more rigid than the aforementioned extended porous polytetrafluoroethylene film, and for the aforementioned reinforcing layer to be obtained by squeezing the pores of the extended porous polytetrafluoroethylene film.

The gasket of the present invention is obtained by forming an appropriate shape from the gasket material of the present invention.

[0009]
[Embodiments of the Invention]

First, the biaxially extended porous polytetrafluoroethylene (hereinafter abbreviated as "biaxial ePTFE") film constituting the gasket material of the present invention will be described.

[0010]

The biaxial ePTFE film used in the present invention is an ePTFE film that is essentially free of nodes whose diameter or major axis exceeds 3 μm per scanning electron microscope (SEM) observation area of 330 μm^2, that is, an ePTFE film in which at least 98% of the nodes have a diameter or major axis of 3 μm. It can be seen that the nodes of the ePTFE film used in the present invention are fairly small because the nodes of ePTFE films used in conventional gasket materials have a diameter or major axis of about 5 μm or greater, and, in extreme cases, reach a size of 400 μm or greater. An ePTFE film virtually devoid of nodes whose diameter or major axis exceeds 3 μm will hereinafter be referred to as "a small-node ePTFE film."

[0011]

In the small-node ePTFE film, the nodes are made infinitely small, and most of the folded crystals are already unraveled and fully extended. The nodes can be distinguished from the fibrils by SEM observation as the nodular portions on the fibrils

(as a mass connecting a plurality of fibrils). The basic structure of a small-node ePTFE film is the same as that of a conventional ePTFE film. Specifically, the film is composed of nodes and fibrils, and a biaxial ePTFE film has a spiderweb, fibrous structure in which the fibrils spread out radially, the nodes that connect the fibrils form isolated islands, and a large number of spaces are defined by the fibrils and nodes (see Figure 9).

[0012]

Such a small-node ePTFE film can be obtained by a process in which two axes are selected to perform stretching when, for example, a sheet obtained by the paste extrusion of PTFE is stretched without being calcined; the stretching ratio is set higher than in the past (specifically, 100%/s or lower, preferably 50%/s or lower, and ideally 20%/s or lower); and the elongation surface factor along the two axes is set to 50 or higher. A more detailed description is given in Japanese Laid-Open Patent Application 7-196831. A semi-calcined article may be used instead of the uncalcined article (Japanese Laid-Open Patent Application 5-202217, etc.).

[0013]

As used herein, the term "stretching rate (%/s)" refers to the ratio of the speed with which two opposing pin frames are being moved away from each other, to the pre-stretching distance between the frames when stretching is performed by moving these two frames away from each other, or to the ratio of the difference in rotational speeds between rollers to the distance between the rollers when stretching is performed between a pair of opposing rollers having different speeds. Furthermore, the term "elongation surface factor" refers to a factor expressed as a product ($\lambda M \times \lambda T$) of the percent of stretch (λM) in the machine direction (MD) and the percent of stretch (λT) in the transverse direction (TD). When stretching is performed by moving opposing pin frames away from each other, "percent of stretch" is defined as a ratio (dimensionless) of the final distance between frames following stretching, to the distance between the frames prior to stretching, or as a ratio (%) of the distance a molded article has been extended (a value obtained by subtracting the initial distance between the frames prior to stretching from the final distance between the frames following stretching) to the initial distance between the frames prior to stretching. When stretching is performed between a pair of opposing rollers having different speeds, percent of stretch is defined as the ratio (dimensionless) of the rotational speeds

of the two rollers or as a ratio of the distance a molded article has been extended (difference in the rotational speed between the two rollers) to the rotational speed of the first roller. For example, a factor of five will thus correspond to 400% when expressed as percent of stretch.

[0014]

The small-node ePTFE film thus obtained commonly has a thickness of about 5 to 200 μm, and its porosity can be appropriately selected in accordance with the percent of stretch from within a range of 40 to 98%, as in the case of a conventional ePTFE film. The average distance between fibrils or the average size of the spaces defined by the fibrils and nodes, that is, the mean pore diameter, can be appropriately set depending on the percent of stretch, preferably to 0.5–5.0 μm, and ideally to 0.5–1.0 μm. This is because excessively large pores will reduce the surface area of contact between films, reduce adhesion between the films, cause permeation leakage when strong fastening pressure is not employed in the system, and compromise rather than improve sealing properties. On the other hand, a mean pore diameter of less than 0.5 μm hampers stretching and fails to produce consistent fiber orientation.

[0015]

Because such small-node ePTFE films have virtually no residual areas of folded crystals, it can be said that the nodes are unraveled and drawn out even when [the gasket] is fastened at a high pressure. This means that no creep occurs.

[0016]

The gasket material of the present invention is a sheet (hereinafter "an ePTFE laminated sheet") obtained by the lamination of a plurality of such small-node ePTFE films. Because the thickness of such small-node ePTFE films is commonly about 5 to 200 μm, it is preferable for about 10 to 500 such sheets to be laminated. Depending on the thickness of the gasket to be obtained, about 100 to 200 sheets are commonly laminated. Although an adhesive may be used to laminate and integrate small-node ePTFE films, it is preferable for such uncalcined small-node ePTFE films to be first laminated and then bonded and integrated by calcining. It is preferable for calcining to be performed at a temperature (specifically, about 350 to 380°C) above the melting point of PTFE (327°C).

desired mechanical properties can be obtained, and the requirements for gasket characteristics met, when a gasket of arbitrary shape is fabricated from the gasket material of the present invention.

[0021]

Specifically, it is preferable for the elongation at break of a small-node ePTFE laminated film used as a gasket to be 200% or lower. (The elongation (%) is measured by a tensile test based on JIS K7113 as the value observed at the moment of rupture of a sample being stretched at a constant rate.) It is also preferable for the difference between the elongation at break in the MD and the elongation at break in TD to be within 20%.

[0022]

Furthermore, it is preferable for the tensile strength of the matrix of the gasket material to be 10 kgf/mm^2 or higher. As used herein, the term "matrix of the gasket material" refers to the node and fibril portion of a biaxial ePTFE laminated sheet (portion that excludes the pores of ePTFE). The tensile strength of the matrix is calculated using the following formula to normalize the results obtained by a tensile test based on JIS K7113 and performed on a sample of a small-node ePTFE laminated film (by measuring the strength achieved at the moment of yield of a sample being stretched at a constant rate).

[0023]

Matrix tensile strength = (Tensile strength) × 2.2/Density,

where "Density" is the apparent density (D = W/V; unit: g/cm^3) calculated by dividing the measured weight (W) of a laminated sheet sample by its volume (V), and "2.2" is the density at a porosity of 0% (density of a PTFE seal obtained by sintering). An increase in the tensile strength of the matrix signifies adequate stretching, which makes creep less likely to occur, and thus prolongs gasket life.

[0024]

Due to the characteristics of small-node ePTFE laminated films, the stress relaxation ratio of the gasket material of the present invention is lower than that of a conventional ePTFE laminated sheet. As used herein, the term "stress relaxation"

refers to a reduction in gasket stress over time due to creep, so the fastening pressure is lowered as a result of stress relaxation. Figure 1 is a graph depicting the relation between the fastening pressure and the amount of leakage created at an internal pressure of 28 kgf/mm^2 in an annular gasket with an outside diameter of 149 mm, an inside diameter of 124 mm, and a thickness of 3.2 mm. It is apparent from Figure 1 that leakage increases with a reduction in fastening pressure. It is preferable for the gasket material pertaining to the present invention (thickness: 3 mm or greater; porosity: 75% or lower) to have a stress relaxation ratio (as determined by a 96-hour ATRS test at 93°C) of 35% or lower, and particularly 25% or lower. This is because a gasket material with a stress relaxation ratio of 35% or lower can provide a gasket capable of preserving its high initial fastening pressure and exhibiting excellent sealing characteristics for a long time.

[0025]

The aforementioned ATRS test is performed using an implement such as that shown in Figure 2. Specifically, a bolt 9 is inserted into a bolt opening formed in a sample 5 shaped as a strip (127 mm × 12.7 mm) and composed of a gasket material, [the sample] is sandwiched between platens 6, and the two ends of this [sample] are fastened with nuts 8 via interposed springs 7 (which reproduce the rigidity of flanges), creating an initial fastening force of 350 kg/cm^2. In such an implement, the bolt 5 is extended when a fastening force (bolt axial force) acts on it, but the bolt becomes proportionately less elongated when the fastening force is reduced. The stress relaxation ratio (%) is calculated as $(D_0 - D_1)/D_0 \times 100$, where D_0 is the elongation of the bolt 9 before the test (during initial fastening), and D_1 is the elongation of the bolt after the test.

[0026]

Another feature of the gasket material of the present invention is that it is preferable for the small-node ePTFE laminated film to have at least one interposed reinforcing layer that is more rigid than the small-node ePTFE film. Figure 3a shows an annular gasket fabricated using a gasket material in which a single reinforcing layer 12 is sandwiched between ePTFE layers 11, and Figure 3b shows an annular gasket fabricated using a gasket material containing two interposed reinforcing layers 12, with the ePTFE layers 11 and reinforcing layers 12 laminated in alternating fashion. Each ePTFE layer 11 is composed of an ePTFE film or a laminate of such

films, and the reinforcing layers 12 are film products composed of a highly rigid material. Laminating the reinforcing layers 12 between the ePTFE layers 11 in such a manner improves the flexural rigidity and strength of the entire gasket, making it easier to handle annular gaskets for large bores. A material that is more rigid than the small-node ePTFE film and that is similar or superior to PTFE in terms of heat resistance should be used for the reinforcing layers 12 in this case. Metal foil, sintered PTFE shaped as films, and products obtained by squeezing the pores of ePTFE films can be cited as examples of materials that satisfy such conditions. Of these, nonporous extended PTFE films obtained by squeezing the pores of ePTFE films are preferred for use because of their excellent adhesion to ePTFE layers. The nonporous extended PTFE films may be conventional ePTFE films or small-node ePTFE films. This is because hard extended PTFE films can be obtained by squeezing the pores. Products obtained by squeezing the pores of small-node ePTFE films are even better because of their rigidity and strength.

[0027]

The gasket of the present invention can be manufactured by punching or cutting the gasket material (small-node ePTFE laminated film) of the present invention to form annular shapes, shapes provided with bolt holes, and other arbitrary shapes. The gasket of the present invention has excellent mechanical properties (and creep resistance in particular), and hence sealing characteristics (and gasket life in particular), because of the characteristics of the gasket material (small-node ePTFE laminated film) of the present invention. In addition, the use of a roughly isotropic gasket material makes it possible to obtain a gasket with the desired physical properties even when a gasket of arbitrary shape is formed from any part of the laminated sheet.

[0028]
[Embodiments]

The present invention will now be described through specific embodiments.

Evaluation Methods

The measurement and evaluation methods used in the embodiments will first be described.

[0029]

(1) Node Size (μm)

Maximum node size (expressed as "Major axis × Minor axis") was determined using SEM photographs (× 5000). Figures 4, 5, and 6 depict SEM photographs of surfaces of the gasket materials (ePTFE laminated sheets) in Embodiment 1 and in Comparative Examples 1 and 2.

[0030]

(2) Porosity (%)

Calculated based on the following formula by making use of the density (2.2) at a porosity of 0% and the density ($D = W/V$; unit: g/cm^3) calculated by dividing the weight (W) of a laminated sheet by its volume (V).

$$\text{Porosity} = \{1 - (D/2.2)\} \times 100$$

[0031]

(3) Stress Relaxation Ratio (%)

ATRS tests were performed at 93°C, 204°C, and 315°C. The initial fastening pressure was set to 350 kgf/mm^2; the fastening time, to 96 hours.

[0032]

(4) Elongation at Break (%)

Tensile tests were performed at normal temperature (23°C) in accordance with JIS K7113. Specifically, No. 2 test pieces (dumbbell-shaped) were fabricated from gasket materials (ePTFE laminated sheets), both ends of each test piece were secured, the test pieces were elongated at a rate of 200 mm/min, and elongation (%) was measured at the moment of rupture of the test pieces.

[0033]

(5) Matrix Tensile Strength (kgf/mm^2)

Tensile tests were performed at normal temperature (23°C) in accordance with JIS K7113. Specifically, No. 2 test pieces (dumbbell-shaped) were fabricated from gasket materials (ePTFE laminated sheets), both ends of each test piece were secured, the test pieces were elongated at a rate of 200 mm/min, and strength (kgf/mm^2) was

measured at the moment of rupture of the test pieces. The result was calculated using the following formula to normalize the measured tensile strength (kgf/mm^2). In the formula, "Density" is the value (D) obtained using the aforementioned porosity.

Matrix tensile strength = Tensile strength × (2.2/Density)

[0034]
(6) Q_p

This value characterizes gasket life and involves determining the stress relaxation ratio calculated as described above and measuring tensile strength in accordance with an ATRS test following standing for 96 hours at 93°C, 204°C, and 315°C. Specifically, samples shaped as strips (127 mm × 12.7 mm) were formed from gasket materials (ePTFE laminated sheets), stretched at a temperature of 23°C and an elastic stress rate of 15 mm/min, and measured using the following formula based on the tensile strength (kgf/mm^2) at the moment of rupture.

(a) When tensile strength is less than 1.75 kgf/mm^2:
Q_p = (Tensile strength/0.7) × {(100 − Stress relaxation ratio/75}^{2}

(b) When tensile strength is 1.75 kgf/mm^2 or higher:
Q_p = 2.5 × {(100 − Stress relaxation ratio/75}^{2}

[0035]
(7) Maximum Service Temperature (°C)

Because Q_p is a function of temperature, Q_p should be calculated at each temperature, and the temperature at which Q_p = 1 should be calculated. Here, Q_p = 1 is a value corresponding to an asbestos joint sheet. The reason that an asbestos joint sheet was used as reference is that it can be used for several tens of years on an actual site and is known to exhibit sufficiently reliable sealing characteristics.

[0036]
(8) Surface Increase Ratio (%)

Annular gasket samples with an outside diameter of 55 mm, an inside diameter of 28 mm, and a thickness of 3 mm were fastened at a fastening pressure of 300 kgf/mm^2 and allowed to stand for 1 hour at a temperature of 200°C. The rings resembled a true circle prior to fastening, as shown in Figure 7a, but creep was caused

by fastening, and deformation occurred (surface area increased in proportion to the reduction in thickness), as shown in Figure 7b. The surface area (S_0) prior to fastening and the surface area (S_1) subsequent to fastening and standing were calculated, yielding the fractional increase in surface area (($S_1 - S_0$) ÷ S_0).

[0037]
(9) Blowout Temperature

Annular gasket samples with an outside diameter of 127 mm, an inside diameter of 89 mm, and a thickness of 3 mm were set in a flange at a fastening pressure of 350 kgf/mm^2, and an internal pressure of 70 kgf/mm^2 was applied. The flange temperature was gradually raised, and the temperature was measured at the moment the gasket samples blew out (the ring ruptured, creating an opening).

[0038]
(10) Amount of Leakage (Initial)

Measured by the ROTT test. Specifically, an annular gasket 23 (outside diameter: 149 mm; inside diameter: 124 mm; thickness: 3 mm) was secured at a fastening pressure of 200 kgf/mm^2 by applying pressure to a fastening member 21 with the aid of a hydraulic pump, as shown in Figure 8, and the system was sealed with the aid of O-rings 22. In this state, helium gas was introduced through an injection port 24 to create a pressure of 70 kgf/mm^2 inside the ring of the annular gasket, and the amount of helium leaking out through a leakage port 25 was measured with a helium leak detector.

[0039]
Fabrication of Gasket Material and Gasket
Embodiment 1

A sheet shape was paste-extruded from a paste product obtained by admixing 17 wt% of solvent naphtha as a lubricant into a PTFE fine powder (manufactured by Daikin), the sheet was then calendered with rolls, and the lubricant was dried off, yielding an uncalcined tape with a thickness of 0.66 mm and a width of 153 mm. This tape was stretched in the MD under conditions corresponding to a stretching rate of 90%/s and a percent of stretch of 7.0 while kept at 300°C. The tape was subsequently stretched in the TD under conditions corresponding to a stretching rate of 85%/s and a percent of stretch of 18.5 while kept at 275°C. The elongation surface factor of the

resulting stretched film in relation to the uncalcined tape corresponded to the product (129.5) of the percent of stretch (7.0) in the MD and the percent of stretch (18.5) in the TD. This film was a small-node ePTFE film with a thickness of 39 μm, a porosity of 86.6%, and a maximum node size of 2 μm × 2 μm. 244 such small-node ePTFE films were superposed and calcined for 60 minutes at 366°C to bond and integrate the films with each other, yielding a laminated sheet with a thickness of 3.66 mm and a porosity of 74.6%.

[0040]
Embodiment 2

A sheet shape was paste-extruded from a paste product obtained by admixing 16 wt% of solvent naphtha as a lubricant into a PTFE fine powder (manufactured by Du Pont), the sheet was then calendered with rolls, and the lubricant was dried off, yielding an uncalcined tape with a thickness of 1.3 mm and a width of 302 mm. This tape was stretched in the MD under conditions corresponding to a stretching rate of 80%/s and a percent of stretch of 9.5 while kept at 300°C. The tape was subsequently stretched in the TD under conditions corresponding to a stretching rate of 95%/s and a percent of stretch of 13.5 while kept at 280°C. The elongation surface factor of the resulting film in relation to the uncalcined tape corresponded to the product (128.3) of the percent of stretch (9.5) in the MD and the percent of stretch (13.5) in the TD. This film was a small-node ePTFE film with a thickness of 52 μm, a porosity of 80.0%, and a maximum node size of 1 μm × 1 μm. 72 such small-node ePTFE films were superposed and calcined for 60 minutes at 365°C to bond and integrate the films with each other, yielding a laminated sheet with a thickness of 2.88 mm and a porosity of 74.6%.

This laminated sheet (gasket material) was punched into annular shapes with an outside diameter of 55 mm and an inside diameter of 28 mm, yielding annular gaskets.

[0041]
Embodiment 3

A sheet shape was paste-extruded from a paste product obtained by admixing 13.wt% of solvent naphtha as a lubricant into a PTFE fine powder (manufactured by Du Pont), the sheet was then calendered with rolls, and the lubricant was dried off, yielding an uncalcined tape with a thickness of 1.3 mm and a width of 302 mm. This

tape was stretched in the MD under conditions corresponding to a stretching rate of 95%/s and a percent of stretch of 9.1 while kept at 300°C. The tape was subsequently stretched in the TD under conditions corresponding to a stretching rate of 90%/s and a percent of stretch of 15.5 while kept at 280°C. The elongation surface factor of the resulting film in relation to the uncalcined tape corresponded to the product (141.1) of the percent of stretch (9.1) in the MD and the percent of stretch (15.5) in the TD. This film was a small-node ePTFE film with a thickness of 62 μm, a porosity of 78.3%, and a maximum node size of 1 μm × 1 μm. 53 such small-node ePTFE films were superposed and calcined for 60 minutes at 365°C to bond and integrate the films with each other, yielding a laminated sheet with a thickness of 3.41 mm and a porosity of 72.3%.

This laminated sheet (gasket material) was punched into annular shapes with an outside diameter of 55 mm and an inside diameter of 28 mm, yielding annular gaskets.

[0042]

Comparative Example 1

A sheet shape was paste-extruded from a paste product obtained by admixing 17 wt% of solvent naphtha as a lubricant into a PTFE fine powder (manufactured by Asahi Glass), the sheet was then calendered with rolls, and the lubricant was dried off, yielding an uncalcined tape with a thickness of 0.24 mm and a width of 152 mm. This tape was stretched in the MD under conditions corresponding to a stretching rate of 150%/s and a percent of stretch of 2.0 while kept at 300°C. The tape was subsequently stretched in the TD under conditions corresponding to a stretching rate of 150%/s and a percent of stretch of 7.0 while kept at 275°C. The elongation surface factor of the resulting film in relation to the uncalcined tape corresponded to the product (14) of the percent of stretch (2.0) in the MD and the percent of stretch (7.0) in the TD. This film was an ePTFE film with a thickness of 45 μm, a porosity of 79.5%, and a maximum node size of 1 μm × 1 μm. 90 such ePTFE films were superposed and calcined for 60 minutes at 365°C to bond and integrate the films with each other, yielding a laminated sheet with a thickness of 3.2 mm and a porosity of 72.7%.

This laminated sheet (gasket material) was punched into annular shapes with an outside diameter of 55 mm and an inside diameter of 28 mm, yielding annular gaskets.

[0043]

Comparative Example 2

A commercially available ePTFE laminated sheet (registered trade name: SEALON; manufactured by Yeu Ming Tai; thickness: 3 mm; porosity: 67.0%) was used as the gasket material. This laminated sheet (gasket material) was punched into annular shapes with an outside diameter of 55 mm and an inside diameter of 28 mm, yielding annular gaskets.

[0044]

The gaskets (or gasket materials) of Embodiments 1 through 3 and Comparative Examples 1 and 2 were measured for matrix tensile strength, elongation at break, stress relaxation ratio, surface increase ratio, blowout temperature, and amount of leakage on the basis of the evaluation method described below. The results are shown in Table 1.

[0045]

[Table 1]

			Embodiments			Comparative Examples	
	No.		1	2	3	1	2
Material	Node size (μm × μm)		2 × 2	1 × 1	1 × 1	5 × 1	5 × 5
	Porosity (%)		74.6	74.6	72.3	72.7	67.0
	Stress relaxation ratio (%)	93°C	25	31	24	37	53
		204°C	41	36	33	57	73
		315°C	78	67	68	90	94
	Elongation at break (%)	MD	120	110	100	320	300
		TD	140	120	100	150	330
	Matrix tensile strength (kgf/mm^2)	MD	15.1	16.0	19.0	5.5	4.9
		TD	14.1	13.8	16.1	9.2	4.9
	Q_p	93°C	2.49	2.13	2.40	1.66	0.98
		204°C	1.56	1.81	2.00	0.83	0.34
		315°C	0.22	0.50	0.45	0.06	0.02
Evaluation	Maximum service temperature (°C)		250	274	276	180	90
	Surface increase ratio (%)		3	0	0	18	26
	Blowout temperature (°C)		341	Unmeasured	Unmeasured	297	282
	Amount of leakage (initial) (atm · cc/sec)		0.017	Unmeasured	Unmeasured	0.0091	0.10

[0046]

Evaluation

As is evident in Table 1, gasket materials having a node major axis of 3 μm or less and gaskets fabricated using such materials (Embodiments 1 through 3) have lower elongation at break, stress relaxation ratio, and matrix tensile strength than conventional ePTFE strips and gaskets fabricated using such sheets (Comparative Examples 1 and 2). Furthermore, the maximum service temperature and the blowout temperature of the former are higher than those of the latter. In terms of initial amount of leakage, Embodiment 1 is superior to Comparative Example 2 but is slightly inferior to Comparative Example 1. However, the stress relaxation ratio, maximum service temperature, and other physical properties are better than in Comparative Example 1, so the reduction in sealing properties (increase in the amount of leakage) is more pronounced in Comparative Example 1 when the gaskets are used at a high temperature or for extended periods at a high fastening pressure, making it possible to anticipate that Embodiment 1 will produce better sealing properties in the long run.

[0047]

Another feature is that the difference between the elongation at break in the MD and the elongation at break in the TD is less than 20% for the gasket materials in Embodiments 1 through 3. In other words, the difference is less than for the gasket materials of the comparative examples, indicating that the materials are isotropic.

[0048]

[Merits of the Invention]

Because the gasket material of the present invention is obtained using an ePTFE film (small-node ePTFE film) that is smaller than a conventional ePTFE film, this material has excellent elongation at break, tensile strength, and other mechanical properties, and hence possesses superior creep resistance. Consequently, a gasket fabricated using the gasket material of the present invention can be used at higher temperatures and fastening pressures than can a gasket fabricated using a conventional film made of ePTFE, and the gasket life is thus prolonged.

[0049]

Another merit is that because a gasket material having an interposed reinforcing layer produces high flexural rigidity in a gasket, annular gaskets for large bores are easier to handle. In addition, products in which reinforcing layers are obtained by squeezing the pores of ePTFE films are 100% PTFE gaskets, and thus preserve the heat and corrosion resistance of PTFE.

[Brief Description of the Drawings]

[Figure 1]

A graph showing the relation between fastening pressure and amount of leakage.

[Figure 2]

A diagram depicting the structure of the implement used in ATRS tests.

[Figure 3]

A diagram depicting the structure of a gasket obtained by the lamination of reinforcing layers.

[Figure 4]

An SEM photograph (× 5000) of the biaxial ePTFE in Embodiment 1 of the present invention.

[Figure 5]

An SEM photograph (× 5000) of the biaxial ePTFE in Comparative Example 1.

[Figure 6]

An SEM photograph (× 5000) of the biaxial ePTFE in Comparative Example 2.

[Figure 7]

A diagram illustrating the method for measuring the surface increase ratio.

[Figure 8]

A diagram depicting the structure of the implement used to measure the amount of leakage.

[Figure 9]

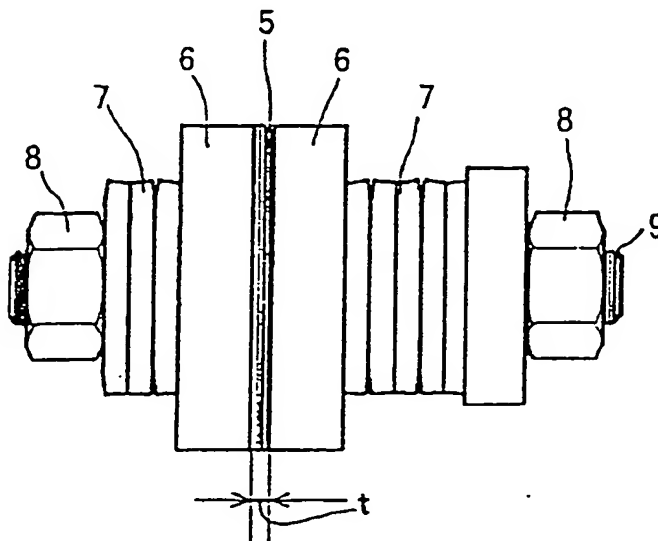A schematic showing the structure of a biaxial ePTFE film.

[Key to Symbols]

1:	fibril
2:	node
3:	pore
5:	gasket material sample
11:	small-node ePTFE film
12:	reinforcing layer
23:	annular gasket

[Document Title] Drawings

[Figure 1]

Size: $\varnothing$ 149 × $\varnothing$ 124 × t 3.2
Internal pressure: 28 kgf/mm^2
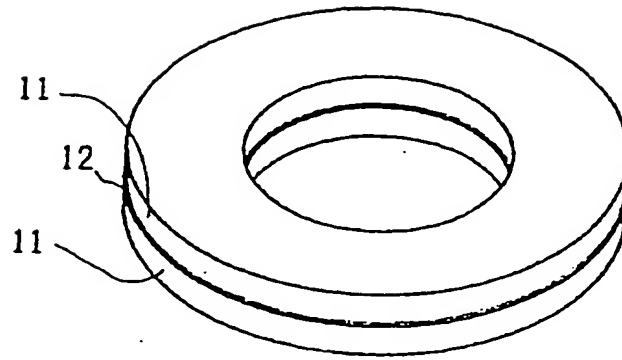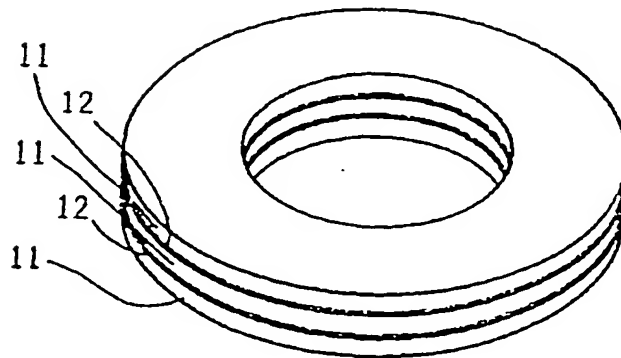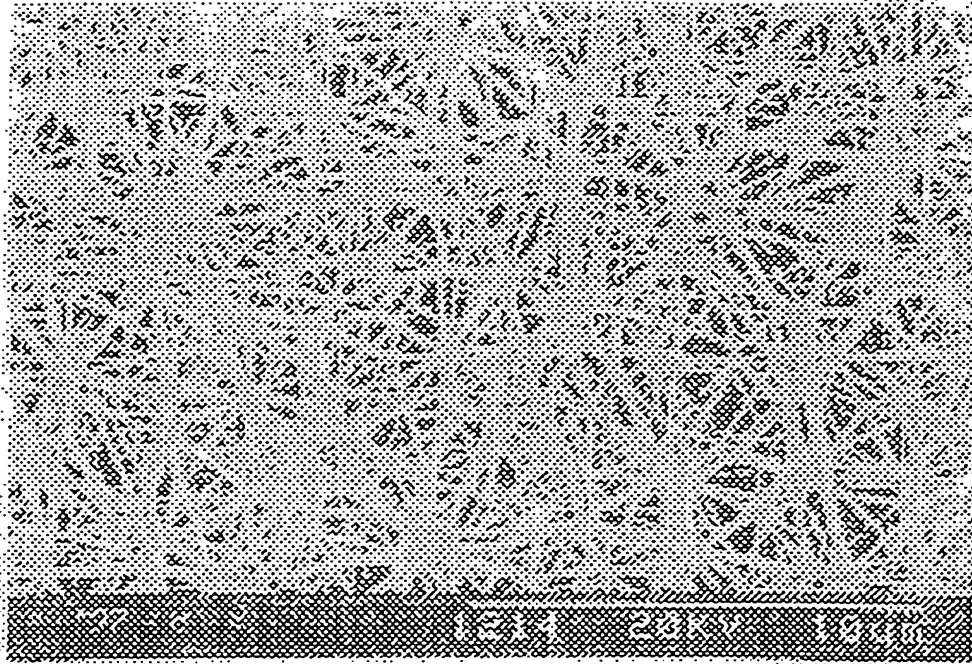
Amount of leakage (atm · cc/sec)

Fastening pressure (kgf/mm^2)

[Figure 2]

[Figure 3]

(a)

11

12

11

(b)

11

12

11

12

11
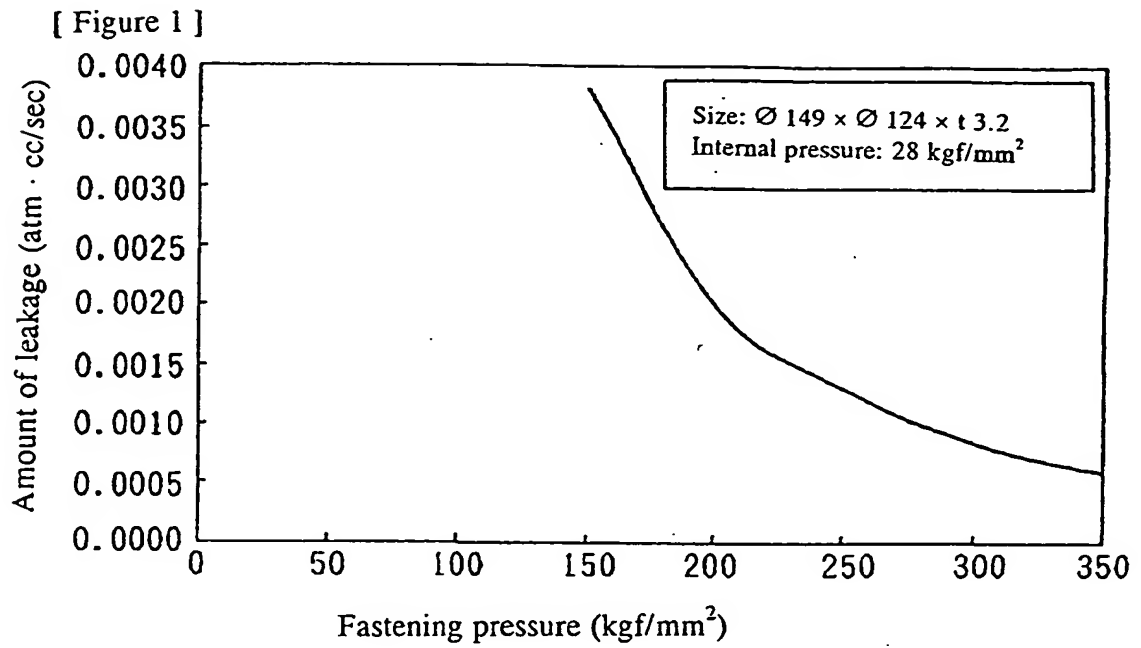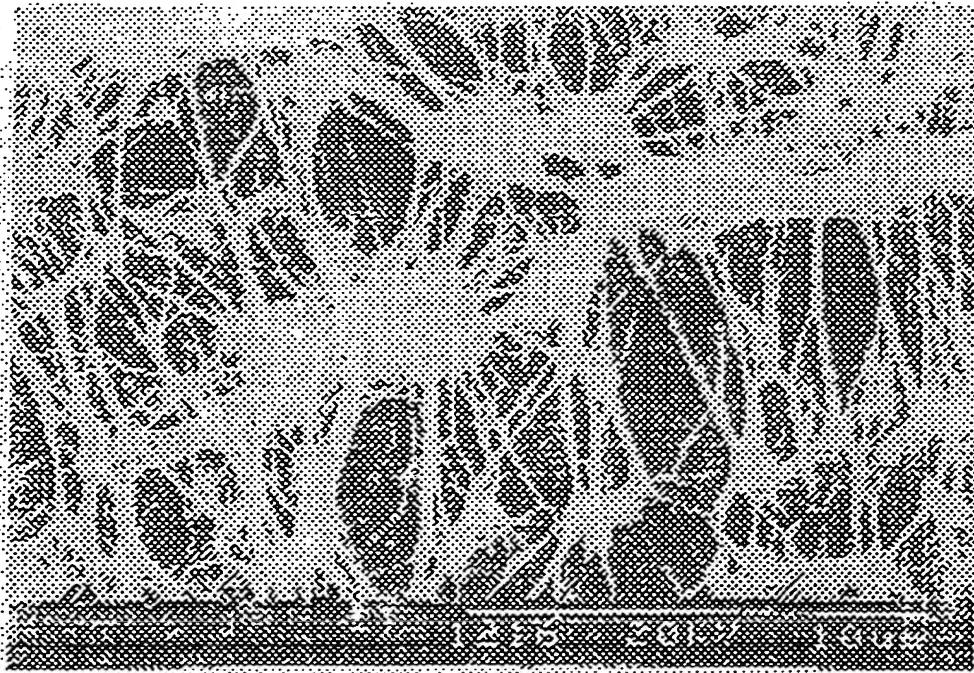
[Figure 4]

[Figure 5]

[Figure 6]
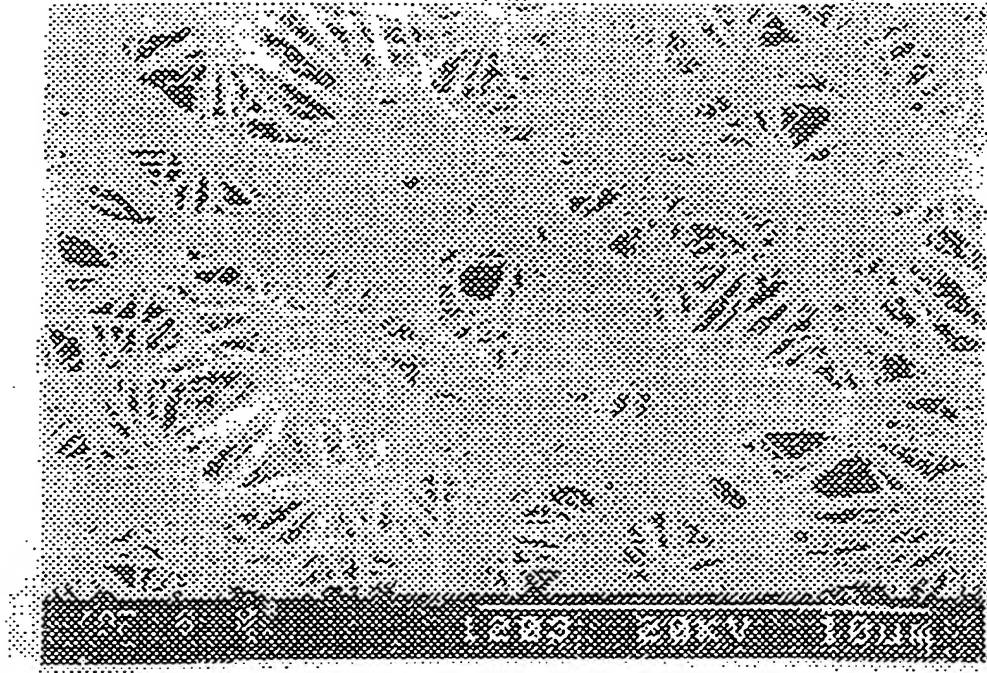
[Figure 7]

(a)
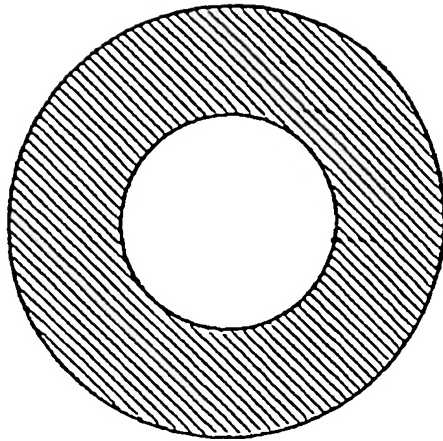
Surface area S_0

(b)

Surface area S_1

[Figure 8]

Hydraulic pump

Measured by helium
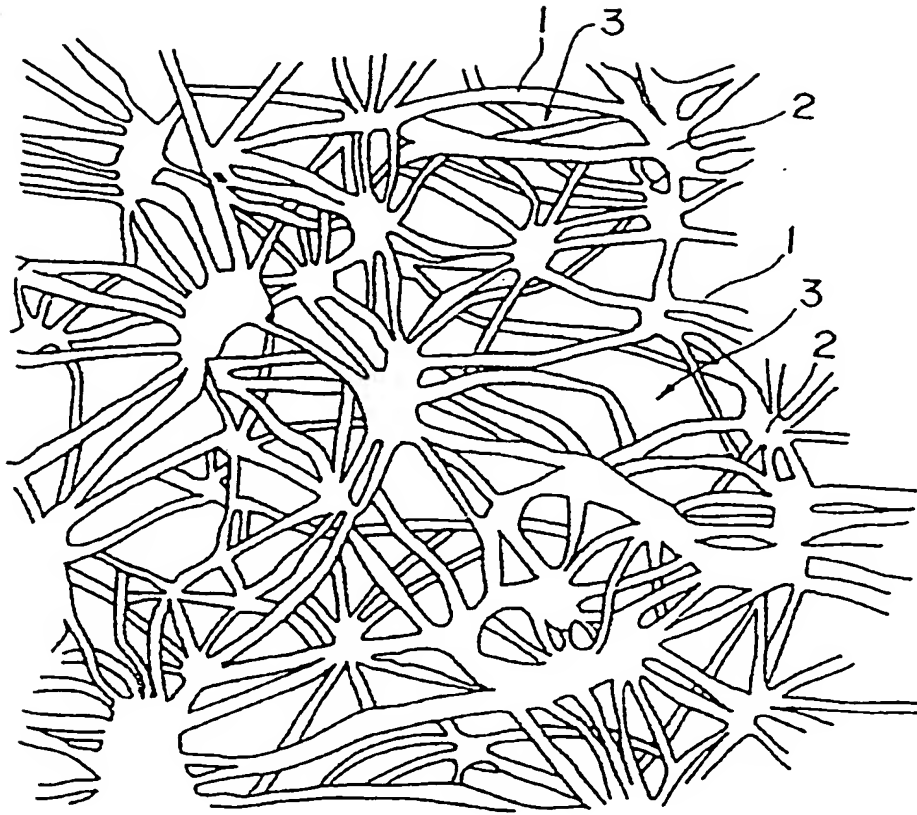detector

He gas

25

24

21

22

23

22

21

Hydraulic pump
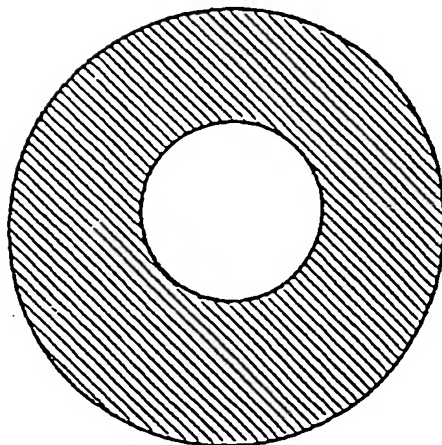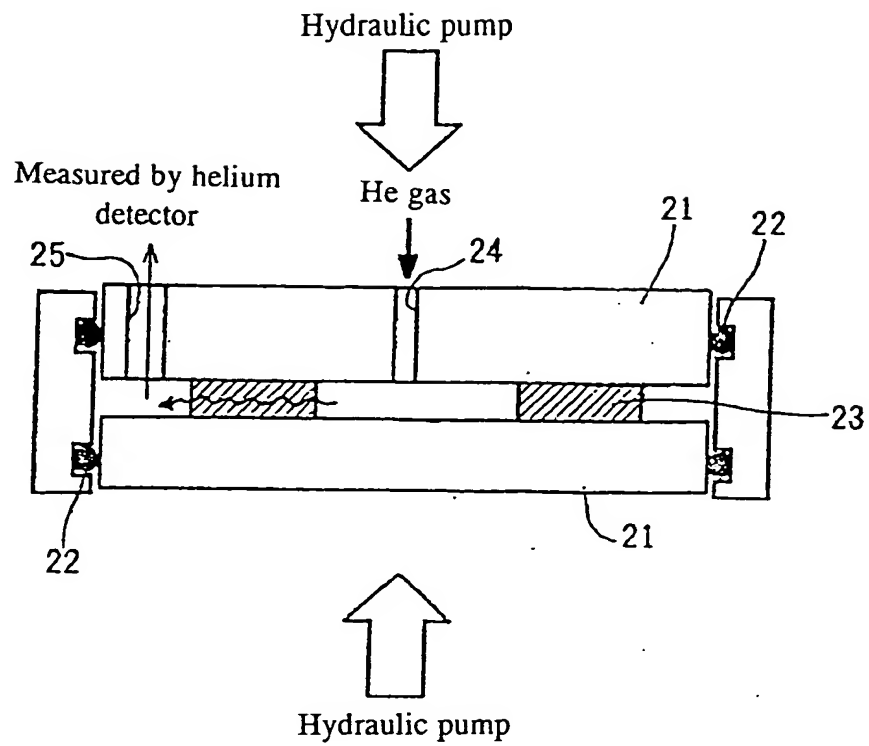
[Figure 9]　　Prior art

[Document Title]   Abstract

[Abstract]

[Object]   To provide an ePTFE gasket that has better creep resistance than in the past, and to provide a gasket material that makes it possible to obtain such a gasket.

[Structure]   A gasket material, composed of a laminated sheet obtained by the lamination of a biaxially extended porous polytetrafluoroethylene film consisting of nodes and of fibrils that connect these nodes, wherein said gasket material is such that the aforementioned biaxially extended porous polytetrafluoroethylene film is essentially free of nodes whose diameter or major axis exceeds 3 μm per scanning electron microscope observation area of 330 μm^2; and a gasket obtained by forming an appropriate shape from the aforementioned gasket material.

[Selected Figure]   Figure 4